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#### SYNTHETIC MAGNESIUM SILICATE COMPOSITIONS AND USES THEREOF

This invention is concerned with synthetic magnesium silicate compositions, methods and uses thereof. In particular, the present invention concerns the use of synthetic magnesium silicate compositions that contain reduced amounts of lithium or are lithium-free, lithium-free synthetic magnesium silicate compositions, methods of extending the gel time of aqueous compositions comprising synthetic magnesium silicate compositions and processes for the preparation of synthetic magnesium silicate compositions.

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With the name "hectorite" has been ascribed to a natural trioctahedral smectite found at Hector, California, USA. This clay is an hydrous magnesium silicate having the ideal composition Si<sub>8</sub> Mg<sub>6</sub> O<sub>20</sub> (OH)<sub>4</sub> modified by having a portion of the Mg<sup>+2</sup> and OH ions replaced by Li+ and F- ions.

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The synthesis of hydrous magnesium silicates similar to natural hectorite has been described by Granquist and Pollack in "Clays and Clay minerals" Vol. 8 (Proceedings of the 8<sup>th</sup> National Conference on Clays and Clay Minerals) pages 150-169. In the process described by Granquist, gels of magnesium hydroxide and of silica are produced separately, are washed, are combined and are re-dispersed in water to form a suspension. Lithium hydroxide or lithium fluoride and sodium hydroxide are added to the suspension that is then treated hydrothermally by refluxing it with stirring until a product having a crystal structure similar to that of hectorite is formed.

While Granquist's product has the crystal structure similar to natural hectorite it does not have good rheological properties. Measuring the Bingham Yield Value of an aqueous dispersion of the substance provides a standard yardstick of rheological properties of a substance. The term Bingham Yield Value (also known as Bingham Yield Stress, these terms being alternatives for the same property) is referred to in standard works on rheology for example in "Rheology Theory and Applications" F R Eirich (Acad. Press) Vol. 1 (1956) page 658 and "Colloidal Dispersions" L K Fisher (N.Y. Bureau of Standards) 2<sup>nd</sup> Edition 1953, pages 150-170 and "The Chemistry and Physics of Clays and other Ceramic Materials" 3<sup>rd</sup> Edition, page463, A B Searle and R W Grimshaw.

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The Bingham Yield Value may be determined by first obtaining a flow curve relating the shear stress to the rate of shear and then extrapolating the straight line section of the curve to the shear stress axis, the intercept being the Bingham Yield Value. It can conveniently be determined on any viscometer capable of measuring a range of shear rates and shear stresses.

The product of Granquist, when in the form of a dispersion obtained using 2g silicate and 100ml tap water, gives a Bingham Yield Value of only about 15 dynes per cm<sup>2</sup>. This is a very low value, inferior to that given by natural hectorite. It also gives a low static gel strength.

Processes for the production of synthetic hydrous magnesium silicate compositions having a crystal structure similar to natural hectorite but having better rheological properties than natural hectorite have been described in GB-A-1054111, GB-A-to1213122 and GB-A-1432770.

The process described in the GB-A-1054111 involves forming a slurry by coprecipitation by slowly combining with heating and agitation in an aqueous medium a constituent providing the magnesium ions with constituents providing the silicon (as silicates), hydroxyl and sodium ions and treating the precipitate hydrothermally to crystallise the synthetic mineral-like clay, washing and dewatering the resulting crystallised product, and drying the product at a temperature up to 450°C. The concentration of the slurry is desirably such that the concentration of the product formed is from 1% to 8% by weight, preferably 4% by weight. The hydrous synthetic magnesium silicate contains fluorine and lithium. The clay-like minerals provided have the structural formula:

$$(Si_8\,Mg_{6\text{-x}}\,Li_x.O_{20}.(OH)_{4\text{-y}}\,F_y)^{x(\text{-})}.^x/_nM^{n(\text{+})}$$

in which x is between 0 and 6, y is from 1 up to but excluding 4, and M is a cation, 30 preferably Na<sup>+</sup>.

The process described in GB-A-1213122 involves precipitating a synthetic magnesium silicate by combining an aqueous solution of a water soluble magnesium salt with an aqueous alkaline solution of one or more sodium compounds in the presence of

dissolved silicon compound and hydrothermally treating the precipitate under pressure to crystallise the synthetic mineral-like clay, separating the resultant solid and liquid phases, washing the resulting crystallised product, and drying the product. The concentration of the precipitate is preferably not more than 5% by weight. The hydrous magnesium silicate product contains no fluorine, optionally contains lithium and has the general formula:

$$[Si_8Mg_aLi_bH_{4+c}O_{24}]^{(12-2a-b-c)-}.M^{(12-2a-b-c)+}$$

where (i) M is a sodium, a lithium or an equivalent of an organic cation, and (ii) the value of a, b, and c is such that either a<6, b>0, c>0, b+c<2, and (a+b+c-6)<2; or a<6, b=0, c,2 and (a+c-6)<2.

The process described in GB-A-1432770 involves the synthesis of an hydrous magnesium silicate having a crystal structure similar to that of hectorite and having the general formula:

15  $[Si_8 (Mg_aLi_bH_c)O_{20}(OH)_{4\nu}F_{\nu}]^{2r}.zM^+$ 

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wherein a is 4.95 to 5.7, b is from 0 to 1.05, c is from 0 to <2, a+b+c is from >4 to <8, y is from 0 to <4, z = 12-2a-b-c, and M is Na<sup>+</sup> or Li<sup>+</sup>. The process comprises the sequential steps of forming an aqueous suspension of magnesium carbonate, forming a silica precipitate in the aqueous suspension magnesium carbonate, the proportions of magnesium provided by the magnesium carbonate and silica precipitated in the suspension corresponding to that of the formula of the magnesium silicate, maintaining the resulting mixture of magnesium carbonate and silica in the wet state and subjecting it to hydrothermal treatment by heating it in an aqueous medium and in the presence of the remaining constituents of the magnesium silicate in proportions within the ranges specified in the general formula and in the presence of excess dissolved sodium or lithium compound over that required to form the cation of the magnesium silicate until crystal growth occurs and separating the resulting crystalline product. The crystalline material resulting from the hydrothermal treatment is then separated by filtration. washed, and dried at a temperature not exceeding 450°C. The process described in GB-A-1432770 is distinguished from the processes described in GB-A-1054111 and GB-A-1213122 in that, in those processes, the Mg compound and the silica are co-precipitated.

Though GB-A-1432770 discloses a general formulation for a synthetic magnesium silicate composition in which there is a possibility for the composition to be lithium-

free, there is no explicit disclosure in GB-A-1432770, or in any other prior art, which indicates a lithium-free composition ever having been prepared.

- The processes described in GB-A-1432770, GB-A-1054111 and GB-A-1213122 are generally batch processes. Continuous processes for the preparation of synthetic magnesium silicates are described in Japanese Patent Application No 06-345419 and in UK Patent Application No 0229630.9, filed 20<sup>th</sup> December 2002.
- The products of the processes described in the above prior art documents are characterised by providing dispersions having Bingham Yield Values substantially in excess of any known to be given by natural hectorite dispersions. Some of these products have found widespread use, by virtue of their excellent rheological properties, in many applications, including in paints and other surface coatings; cosmetic products; horticulture; shampoos; detergents; disinfectants; toothpastes; paper manufacture, for example as fillers, retention and drainage aids, and in paper coatings; and drilling muds. The products of the above batch processes are commercially available as dry white powders, such as the products sold by Rockwood Additives Limited, England, under the trademark "LAPONITE" and, when fully dispersed and hydrated in water, the resulting composition is colourless and transparent.

As mentioned above, synthetic magnesium silicate compositions are characterised by providing dispersions having Bingham Yield Values substantially in excess of any known natural hectorite dispersions, and have resulted in their use, by virtue of their excellent rheological properties, in various applications including in surface coatings. The free flowing white powder takes no more than an hour at ambient temperatures from it being dispersed under shear in water at a level from 1% by wt to hydrate sufficiently to form a highly viscous, thixotropic gel. The time required to form the gel can be reduced by using increased temperatures and by using higher shearing conditions.

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It is known, for example from an inspection of the www.laponite.com website, that at concentrations of above 2% by wt synthetic magnesium silicate in tap water, highly thixotropic gels are formed(substantially less than one hour e.g a 5% by wt aqueous

composition may gel in 5 minutes), which can make it difficult to incorporate other raw materials into the formulation. Accordingly, if the concentration of synthetic magnesium silicate to be used is above 2% by wt, the use of a degelling agent, such as tetrasodium pyrophosphate or a low molecular weight polyethylene glycol, is often recommended.

Whilst the onset of gelling can be delayed by the presence of a degelling agent, some applications of the end formulation, such as coatings for some metals, do not permit the presence of condensed phosphates. Further, it may be more convenient in a manufacturing process not to have to rely upon the presence of a degelling agent.

It is an object of the present invention to delay the onset of gelling in aqueous formulations comprising synthetic magnesium silicate compositions. It is another object to provide synthetic magnesium silicate compositions which demonstrate delayed gelling properties. It is another object of the present invention to reduce or eliminate the amount of degelling agent e.g. phosphate used in an aqueous synthetic magnesium silicate compositions. Preferably, the delay in gelling will occur without having any detrimental effects on the final properties of the gelled composition.

In accordance with a first aspect of the present invention, there is provided the use of a non-hydrated synthetic magnesium silicate compostion of Formula I below:

#### Formula I

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$$[Si_8 (Mg_aLi_bH_c)O_{20}(OH)_{4-v}F_v]^{z-z}/_nM^{n+z}$$

wherein a is 4.95 to 6.6, b is from 0 to 1.5, c is from 0 to <2, a+b+c is from 5 to <8, y is from 0 to <4, z = 12-2a-b-c, M is a cation which is other than Li, and n is the valency of M, wherein the ratio of Si:Mg is from 8:6.6 to 8:4.95 in a method for the preparation of an aqueous composition comprising said synthetic magnesium silicate composition in a hydrated form, which method comprises adding under mixing conditions said non-hydrated synthetic magnesium silicate composition to an aqueous fluid and allowing said non-hydrated synthetic magnesium silicate composition to hydrate.

In another aspect, the present invention provides the use of a non-hydrated synthetic magnesium silicate composition of Formula I below:

Formula I

$$[Si_8 (Mg_aLi_bH_c)O_{20}(OH)_{4-y}F_y]^{z-z}/_nM^{n+z}$$

wherein a is 4.95 to 6.6, b is from 0 to 1.5, c is from 0 to <2, a+b+c is from 5 to <8, y is from 0 to <4, z = 12-2a-b-c, M is a cation which is other than Li, and n is the valency of M, wherein the ratio of Si:Mg is from 8:6.6 to 8:4.95in a method for the preparation of a gelled aqueous composition comprising said synthetic magnesium silicate composition in a hydrated form, which method comprises adding under mixing conditions said non-hydrated synthetic magnesium silicate composition to an aqueous fluid and allowing said non-hydrated synthetic magnesium silicate composition to hydrate, for delaying gelling of said gelled aqueous composition.

In another aspect, the present invention provides a method of reducing or eliminating the amount of degelling agent used in the preparation of an aqueous composition comprising a synthetic magnesium silicate composition in hydrated form, which method comprises adding under mixing conditions a non-hydrated synthetic magnesium silicate composition of Formula I:

Formula I

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$$[Si_8 (Mg_aLi_bH_c)O_{20}(OH)_{4-y}F_y]^{z-}.^{z}/_nM^{n+}$$

wherein a is 4.95 to 6.6, b is from 0 to 1.5, c is from 0 to <2, a+b+c is from 5 to <8, y is from 0 to <4, z = 12-2a-b-c, M is a cation which is other than Li, and n is the valency of M, wherein the ratio of Si:Mg is from 8:6.6 to 8:4.95

to an aqueous fluid and allowing said non-hydrated synthetic magnesium silicate composition to hydrate.

In another aspect, there is provided an aqueous composition comprising an aqueous fluid and a synthetic magnesium silicate composition of Formula I:

Formula I

$$[Si_8 (Mg_aLi_bH_c)O_{20}(OH)_{4-y}F_y]^{z-z}/_nM^{n+z}$$

wherein a is 4.95 to 6.6, b is from 0 to 1.5, c is from 0 to <2, a+b+c is from 5 to <8, y is from 0 to <4, z = 12-2a-b-c, M is a cation which is other than Li, and n is the valency of M, wherein the ratio of Si:Mg is from 8:6.6 to 8:4.95

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Preferably, the synthetic magnesium silicate composition used in the preset invention is completely free of Li. Such a composition preferably has the composition of Formula II below:

Formula II

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$$[Si_8 (Mg_aH_c)O_{20}(OH)_{4-v}F_v]^{z-z}/_nM^{n+}$$

wherein a is 4.95 to 6.6, c is from 0 to <2, a+c is from 5 to <8, y is from 0 to <4, z=12-2a-c, M is a cation which is other than Li, and n is the valancy of M, wherein the ratio of Si:Mg is from 8:6.6 to 8:4.95

In accordance with another aspect of the present invention there is provided a synthetic magnesium silicate composition of Formula II below:

Formula II

$$[Si_8 (Mg_aH_c)O_{20}(OH)_{4-v}F_v]^{z-z}/_nM^{n+z}$$

wherein a is 4.95 to 6.6, c is from 0 to <2, a+c is from 5 to <8, y is from 0 to <4, z = 12-2a-c, M is a cation which is other than Li, and n is the valancy of M, wherein the ratio of Si:Mg is from 8:6.6 to 8:4.95

In the synthetic magnesium silicate compositions of Formula I and Formula 2:

- a) the Si:Mg ratio is prefereably from 8:5.8 to 8:5.3, more preferably from 8:5.68 to 8:5.42;
- b) the M<sup>+</sup> cation is preferably Na<sup>+</sup>;
- c) y is preferably from 0 to 3.

It has been found that by reducing or eliminating the amount of Li in the synthetic

magnesium silicate composition and employing a specific Si:Mg ratio, the time within which the composition gels once it has been added to water or other aqueous fluid is extended. For example, it can be demonstrated that an aqueous composition formed by the addition to water under conventional mixing conditions at room temperature of a synthetic magnesium silicate composition of Formula II in an amount of 5 wt % may take well over an hour before it forms a highly viscous, gelled composition. Further, once the composition has gelled, its rheological properties are substantially similar to the aqueous gels formed from hydrating conventional high-content lithium magnesium silicate compositions. This finding is very surprising and, at present, cannot be explained by the inventors.

The ability to delay gelling without loss of final gel properties offers significant advantages over the conventional compositions. For example, it enables the preparation of aqueous compositions comprising higher amounts of synthetic magnesium silicate compositions than would normally be possible with conventional lithium containing magnesium silicates. Further, aqueous compositions can be prepared which contain reduced or no degelling agents e.g. phosphate free coatings may now be available for use in the metal finishing industry.

- The compositions of Formula I may be prepared by the processes detailed in GB-A-1054111, GB-A-to1213122, GB-A-1432770, Japanese Patent Application No 06-345419 and UK Patent Application No 0229630.9, which are incorporated herein by reference.
- The compositions of Formula II may be prepared by a similar procedure as detailed in GB-A-1054111, GB-A-to1213122, GB-A-1432770, Japanese Patent Application No 06-345419 and UK Patent Application No 0229630.9, except that all Li-containing compounds are absent. Accordingly, in another aspect, the present invention provides a method of preparing a synthetic magnesium silicate composition of Formula II, having a crystal structure similar to natural hectorite, wherein the process comprises the steps of a) forming a precursor slurry by mixing and reacting together water, magnesium sulphate, sodium carbonate and sodium silicate, b) subjecting said precursor slurry to a hydrothermal reaction at a temperature of from 210 to 400°C, and c) washing and filtering to remove water soluble salts formed in the preparation of the precursor slurry.

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The composition of Formula II retains substantially similar rheological properties to the products formed in GB-A-1054111, GB-A-1213122 and GB-A-1432770. The compositions may be supplied as dry white powders or as moist solids or in dispersions. Accordingly, the compositions may be used in the same type of applications as the prior art products. For example, the compositions may be used in paints; cosmetic products; horticulture; shampoos; detergents; disinfectants; toothpastes; paper manufacture, for example as fillers, retention and drainage aids, and in paper coatings; and drilling muds. The Li-free compositions of Formula II, may additionally be useful in applications in

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which the product is intended to be consumed, such as in food and medicinal applications.

In yet another aspect, the present invention provides a method of extending the gelling time of an aqueous composition comprising a synthetic magnesium silicate composition in hydrated form and a degelling agent, such as a glycol or a phosphate, which method comprises adding under mixing conditions a non-hydrated synthetic magnesium silicate having a structure similar to natural hectorite, preferably as made by any of the processes claimed in GB-A-1432770, GB-A-1054111, GB-A-1213122, Japanese Patent Application No 06-345419 and in UK Patent Application No 0229630.9 (filed 20<sup>th</sup> December 2002), to an aqueous fluid and allowing said non-hydrated synthetic magnesium silicate composition to hydrate, characterized in that the aqueous fluid into which said non-hydrated composition is added comprises at least 80% by weight, preferably 100 % by weight, of the degelling agent used in the aqueous composition. In one particular embodiment, the degelling agent is a glycol, which is preferably selected from one or more of ethylene glycol, propylene glycol, polyethylene glycol and polypropylene glycol. Preferably, the non-hydrated synthetic magnesium silicate composition is of Formula I:

Formula I

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$$[Si_8 (Mg_aLi_bH_c)O_{20}(OH)_{4-v}F_v]^{z-z}/_nM^{n+z}$$

wherein a is 4.95 to 6.6, b is from 0 to 1.5, c is from 0 to <2, a+b+c is from 5 to <8, y is from 0 to <4, z=12-2a-b-c, M is a cation which is other than Li, and n is the valency of M, wherein the ratio of Si:Mg is from 8:6.6 to 8:4.95. More preferably, the synthetic magnesium silicate composition is of Formula II below:

25 Formula II

$$[Si_8 (Mg_aH_c)O_{20}(OH)_{4-v}F_v]^{z-.z}/_nM^{n+}$$

wherein a is 4.95 to 6.6, c is from 0 to <2, a+c is from 5 to <8, y is from 0 to <4, z = 12-2a-c, M is a cation which is other than Li, and n is the valancy of M, wherein the ratio of Si:Mg is from 8:6.6 to 8:4.95.

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The following examples illustrate aspects of the invention, but are not in any way intended to limit the scope the invention.

Various synthetic magnesium silicate compositions were prepared by the procedure detailed in Examples 1 and 2 below from the amounts of raw materials, as indicated in Table 1:

#### 5 Example 1 – Preparation of precursor slurry (bulk preparation)

A measured quantity of magnesium sulphate and water (sufficient to dissolve the measured quantity of magnesium carbonate) is placed in a flask fitted with a stirrer, a heating mantle and a refluxing condenser. The mixture was brought to a temperature of at least 60°C under reflux while stirring efficiently.

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From a separate vessel a measured quantity of sodium carbonate solution is added slowly to the reaction vessel containing the magnesium sulphate solution. The addition is made over a period of up to one hour, while the reaction mixture is kept at 60°C or greater and stirred efficiently throughout.

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From a separate vessel a measured quantity of sodium silicate solution is added slowly to the reaction vessel containing the magnesium sulphate and sodium carbonate solution. The addition is made over a period of up to one hour, while the reaction mixture is kept at 60°C or greater and stirred efficiently throughout.

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The mixture is then boiled under reflux, with efficient stirring, for about 2 hours.

#### Example 2 – Hydrothermal treatment (bulk preparation)

The precursor slurry obtained in each of Example 1 was fed into a reactor having an internal temperature of 200°C and pressure of 16 barg. The slurry now undergoes a hydrothermal reaction. The material is retained in the reactor for up to 18 hours, before it is ejected from the reactor into a bath where magnesium silicate crystal so formed are wash and filtered to remove soluble salts.

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After being drying at 105°C and milled to particle size of no greater than 250 microns,

the rheology properties of the powders so produced were evaluated. The results of the evaluation are also presented in Table 1.

From Table 1, it is evident that compositions comprising reduced or no lithium develop gelling properties more slowly than the conventional composition Laponite RD.

Laponite RD is a synthetic magnesium silicate of the typical formula

5 [Si<sub>8</sub>(Mg<sub>5.5</sub>Li<sub>0.3</sub>H<sub>0.2</sub>)O<sub>20</sub>(OH)<sub>4</sub>]<sup>0.5</sup>-<sub>0.5</sub>Na<sup>+</sup>, available from Rockwood Additives Limited, England.

Viscosity was measured at 25°C using a number 4 Ford viscosity cup. The unit of measurement for this method is 'seconds'.

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Table 1

		Composi	Composition (grams)	(8	Si:Mg	Vis	cosity (49	Viscosity (4% dispersion in water)	n in wate	(F
	Li <sub>2</sub> CO <sub>3</sub>	MgSO <sub>4</sub>	Na <sub>2</sub> CO <sub>3</sub>	Na silicate*		0 hour	1 hour	24 hours	3 days	7days
Laponite RD	2.24	100.02	82.77	72.75	8:5.5	20	gelled	gelled	gelled	gelled
Sample 1	0	120.24	87.39	72.75	8:6.58	38	gelled	gelled	gelled	gelled
Sample 2	0	112.95	87.39	72.75	8:6.19	26	09<	gelled	gelled	gelled
6	ŀ	10001					ļ		1	
Sample 3	9	109.31	87.39	72.75	8:6.01	21	09<	gelled	gelled	gelled
Sample 4	0	104.75	88.55	72.75	8:5.82	11	13	20	09<	gelled
Sample 5	0	102.00	88.61	72.75	8:5.6	11	=	12	15	35
Sample 6	0	95.19	87.71	72.75	8:5.32	11	13	gelled	gelled	gelled
								Ď.		0

\* the nominal weight of the SiO<sub>2</sub> content of the solution used is shown in this column

Table 2A

									13	<u>.,</u>	
	2 days	23	Gelled	Gelled	Gelled	Gelled	11	15	09<	Gelled	Gelled
in deionised water)	24 hours	14	Gelled	Gelled	Gelled	Gelled	11	12	09<	09<	Gelled
Viscosity (dispersed in deionised water)	1 hour	11	13	Gelled	Gelled	Gelled	11	11	28	28	09<
	0 hour	11	12	09<	Gelled	Gelled	11	11	15	15	31
%w/w synthetic silicate		2	4	9	8	10	2	4	9	8	10
		Laponite RD					Sample 5				

Table 2A demonstrates that, when used at the same concentration, compositions of the present invention do not gell as quickly as the commercially available Laponite RD material.

Table 2B

opylene glycol)	2 days	Gelled	Gelled	Gelled	12	09<	Gelled
containing 3.5% polypro	24 hours	Gelled	Gelled	Gelled	11	51	Gelled
Viscosity (dispersed in deionised water containing 3.5% polypropylene glycol)	1 hour	09<	Gelled	Gelled		14	09<
Viscosity (dispe	0 hour	24	Gelled	Gelled		12	28
%w/w synthetic silicate		9	00	10	9	∞	10
		Laponite RD			Sample 5		

Table 2A demonstrates that compositions of the present invention can be dispersed at higher concentrations than the commercially available Laponite RD material without undergoing substantially immediate gelling.

Claims

1. The use of a non-hydrated synthetic magnesium silicate composition of Formula I below:

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#### Formula I

$$[Si_8 \, (Mg_aLi_bH_c)O_{20}(OH)_{4\text{-y}}F_y]^{z\text{-}.z}/_nM^{n\text{+}}$$

wherein a is 4.95 to 6.6, b is from 0 to 1.5, c is from 0 to <2, a+b+c is from 5 to <8, y is from 0 to <4, z = 12-2a-b-c, M is a cation which is other than Li, and n is the valency of M, wherein the ratio of Si:Mg is from 8:6.6 to 8:4.95 in a method for the preparation of an aqueous composition comprising said synthetic magnesium silicate composition in a hydrated form, which method comprises adding under mixing conditions said non-hydrated synthetic magnesium silicate composition to an aqueous fluid and allowing said non-hydrated synthetic magnesium silicate composition to hydrate.

2. The use of a non-hydrated synthetic magnesium silicate composition of Formula I below:

Formula I

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## $[Si_8 (Mg_aLi_bH_c)O_{20}(OH)_{4-y}F_v]^{z-z}/_nM^{n+}$

wherein a is 4.95 to 6.6, b is from 0 to 1.5, c is from 0 to <2, a+b+c is from 5 to <8, y is from 0 to <4, z = 12-2a-b-c, M is a cation which is other than Li, and n is the valency of M, wherein the ratio of Si:Mg is from 8:6.6 to 8:4.95

- in a method for the preparation of a gelled aqueous composition comprising said

  25 synthetic magnesium silicate composition in a hydrated form, which method comprises
  adding under mixing conditions said non-hydrated synthetic magnesium silicate
  composition to an aqueous fluid and allowing said non-hydrated synthetic magnesium
  silicate composition to hydrate, for delaying gelling of said gelled aqueous composition.
- 30 3. A method of reducing or eliminating the amount of degelling agent used in the preparation of an aqueous composition comprising a synthetic magnesium silicate composition in hydrated form, which method comprises adding under mixing conditions a non-hydrated synthetic magnesium silicate composition of Formula I:

Formula I

$$[Si_8 (Mg_aLi_bH_c)O_{20}(OH)_{4-v}F_v]^{z-z}/_nM^{n+}$$

wherein a is 4.95 to 6.6, b is from 0 to 1.5, c is from 0 to <2, a+b+c is from 5 to <8, y is from 0 to <4, z = 12-2a-b-c, M is a cation which is other than Li, and n is the valency of M, wherein the ratio of Si:Mg is from 8:6.6 to 8:4.95

- to an aqueous fluid and allowing said non-hydrated synthetic magnesium silicate composition to hydrate.
- 4. An aqueous composition comprising an aqueous fluid and a synthetic
   10 magnesium silicate composition of Formula I:

Formula I

$$[Si_8 (Mg_aLi_bH_c)O_{20}(OH)_{4-v}F_v]^{z-z}/_nM^{n+}$$

wherein a is 4.95 to 6.6, b is from 0 to 1.5, c is from 0 to <2, a+b+c is from 5 to <8, y is from 0 to <4, z = 12-2a-b-c, M is a cation which is other than Li, and n is the valency of

- 15 M, wherein the ratio of Si:Mg is from 8:6.6 to 8:4.95
  - 5. The use of claim 1 or claim 2, method of claim 3 or composition of claim 4, wherein the synthetic magnesium silicate composition is free of Li.
- 20 6. The use of claim 1 or claim 2, method of claim 3 or composition of claim 4, wherein the non-hydrated synthetic magnesium silicate composition is of Formula II below:

Formula II

$$[Si_8 (Mg_aH_c)O_{20}(OH)_{4-v}F_v]^{2-z}/_nM^{n+}$$

- wherein a is 4.95 to 6.6, c is from 0 to <2, a+c is from 5 to <8, y is from 0 to <4, z = 12-2a-c, M is a cation which is other than Li, and n is the valancy of M, wherein the ratio of Si:Mg is from 8:6.6 to 8:4.95.
- 7. The use, method, or composition as claimed in any one of the preceding claims, wherein the aqueous composition comprises a degelling agent.
  - 8. The use, method or composition of claim 7, wherein the degelling agent is a glycol, preferably ethylene glycol, propylene glycol or a low molecular weight polyglycol.

9. A synthetic magnesium silicate composition of Formula II below: Formula II

## $[Si_8 (Mg_aH_c)O_{20}(OH)_{4-y}F_y]^{z-.z}/_nM^{n+}$

- wherein a is 4.95 to 6.6, c is from 0 to <2, a+c is from 5 to <8, y is from 0 to <4, z = 12-2a-c, M is a cation which is other than Li, and n is the valancy of M, wherein the ratio of Si:Mg is from 8:6.6 to 8:4.95
- 10. A method of extending the gelling time of an aqueous composition comprising a synthetic magnesium silicate composition in hydrated form and degelling agent, preferably a glycol, which method comprises adding under mixing conditions a non-hydrated synthetic magnesium silicate having a structure similar to natural hectorite, preferably as made by any of the processes claimed in GB-A-1432770, GB-A-1054111, GB-A-1213122, Japanese Patent Application No 06-345419 and in UK Patent
  15 Application No 0229630.9 (filed 20<sup>th</sup> December 2002), to an aqueous fluid and allowing said non-hydrated synthetic magnesium silicate composition to hydrate, characterized in that the aqueous fluid into which said non-hydrated composition is added comprises at least 80% by weight, preferably 100 % by weight, of the degelling agent used in the aqueous composition.
  - 11. The method of claim 10, wherein the degelling agent is a glycol, preferably a glycol selected from one or more of ethylene glycol, propylene glycol, polyethylene glycol and polypropylene glycol.
- 25 12. The method of claim 11 or claim 12, wherein the non-hydrated synthetic magnesium silicate composition is of Formula I:

#### Formula I

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$$[Si_8 \ (Mg_aLi_bH_c)O_{20}(OH)_{4\text{-y}}F_y]^{z\text{-}}.^{2}/_nM^{n+}$$

wherein a is 4.95 to 6.6, b is from 0 to 1.5, c is from 0 to <2, a+b+c is from 5 to <8, y is
from 0 to <4, z = 12-2a-b-c, M is a cation which is other than Li, and n is the valency of
M, wherein the ratio of Si:Mg is from 8:6.6 to 8:4.95.

13. The method of claim 12, wherein the synthetic magnesium silicate composition is of Formula II below:

Formula II

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 $[Si_8 (Mg_aH_c)O_{20}(OH)_{4-y}F_y]^{z-.z}/_nM^{n+}$ 

wherein a is 4.95 to 6.6, c is from 0 to <2, a+c is from 5 to <8, y is from 0 to <4, z=122a-c, M is a cation which is other than Li, and n is the valancy of M, wherein the ratio
of Si:Mg is from 8:6.6 to 8:4.95.

- 14. The use, method, or composition as claimed in any one of the preceding claims, wherein the Si:Mg ratio in the synthetic magnesium silicate composition is from 8:5.8 to 8:5.3, preferably from 8:5.68 to 8:5.42.
- 15. The use, method, or composition as claimed in any one of the preceding claims, wherein the M<sup>+</sup> cation in the synthetic magnesium silicate composition is Na<sup>+</sup>
- 16. The use, method, or composition as claimed in any one of the preceding claims, wherein y in the synthetic magnesium silicate composition is from 0 to 3.